Rec'd PCT/PTO 22 MAR 2001

FORM PTO-1390 (Modified) (REV 5-93) U S DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER
37838-0009

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371

U.S APPLICATION NO. (If known, see 37 C.F.R. 1.5)
09/787416

INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/EP99/06820	September 15, 1999	September 24, 1998

TITLE OF INVENTION
METHOD AND APPARATUS FOR THE MANUFACTURE OF A TUBE MADE OF FILM ON A CELLULOSE BASIS WHICH REINFORCES AN INSERT

APPLICANT(S) FOR DO/EO/US
Herbert Gord, Klaus-Dieter Hammer, Helmut Sattler, and Rainer Neeff

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. X This is a FIRST submission of items concerning a filing under 35 U.S.C. 371.
2. ___ This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371.
3. ___ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. X A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. X A copy of the International Application as filed (35 U.S.C. 371(c)(2))
   a. ___ is transmitted herewith (required only if not transmitted by the International Bureau).
   b. X has been transmitted by the International Bureau.
   c. ___ is not required, as the application was filed in the United States Receiving Office (RO/US)
6. X A translation of the International Application into English (35 U.S.C. 371 (c)(2)).
   a. X is attached hereto.
   b. ___ has been previously submitted under 35 U.S.C. 154(d)(4).
7. X Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
   a. ___ are transmitted herewith (required only if not transmitted by the International Bureau).
   b. ___ have been transmitted by the International Bureau.
   c. ___ have not been made; however, the time limit for making such amendments has NOT expired.
   d. X have not been made and will not be made.
8. ___ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ___ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ___ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern other document(s) or information included:

11. X An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ___ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. X A FIRST preliminary amendment.
    ___ A SECOND or SUBSEQUENT preliminary amendment.
14. X A substitute specification.
15. ___ A change of power of attorney and/or address letter.
16. X Other items or information: PCT/IB/308

JC03 Rec'd PCT/PTO 22 MAR 2001

U.S. APPLICATION NO. (If known, see 37 C F R. 1 50)	INTERNATIONAL APPLICATION NO.	ATTORNEY'S DOCKET NUMBER
09/787416	PCT/EP99/06820	37838-0009

17. X The following fees are submitted: **CALCULATIONS** PTO USE ONLY

Basic National Fee (37 CFR 1.492(a)(1)-(5):

Neither international preliminary examination fee (CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO.......... **$1000.00**

International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO . **$860.00**

International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO............. **$710.00**

International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4).................. **$690.00**

International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4)........................ **$100.00**

ENTER APPROPRIATE BASIC FEE AMOUNT =				$ 860.00	
Surcharge of $130.00 for furnishing the oath or declaration later than 20 ___ 30 ___ months from the earliest claimed priority date (37 CFR 1.492(e))				$	
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total Claims	25 -20 =	5	X $18.00	$ 90.00	
Independent Claims	2 -3 =	0	X $80.00	$ 0	
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			+ $270.00	$	
TOTAL OF ABOVE CALCULATIONS =				$ 950.00	
Reduction by ½ for filing by small entity, if applicable. Verified Small Entity statement must also be filed. (Note 37 CFR 1.9, 1.27, 1.28).				$	
SUBTOTAL =				$ 950.00	
Processing fee of $130.00 for furnishing English translation later the 20 ___ 30 ___ months from the earliest claimed priority date (37 CFR 1.492(f). +				$	
TOTAL NATIONAL FEE =				$ 950.00	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). **$40.00** per property +				**$**	
TOTAL FEES ENCLOSED =				**$ 950.00**	
				Amount to be: refunded	$
				charged:	$

a. X A check in the amount of $950.00 to cover the above fees is enclosed.

b. ___ Please charge my Deposit Account No. ___ in the amount of $_____ to the above fees. A duplicate copy of this sheet is enclosed.

c. X The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 08-1641. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

Susan E. Shaw McBee
Heller Erhman White McAuliffe
815 Connecticut Avenue, N.W., Suite 200
Washington, D.C. 20006-4004
Tel: (202) 263-8900
Fax: (202) 785-8877

26633
PATENT TRADEMARK OFFICE

Susan E. Shaw McBee
SIGNATURE

Susan E. Shaw McBee
NAME

39,294
REGISTRATION NUMBER

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Attorney Docket No: 37838-0009

Applicants: Herbert GORD *et al.*

Application No.: 09/787,416

Filing Date: June 15, 2001

Title: METHOD AND APPARATUS FOR THE MANUFACTURE OF A TUBE MADE OF FILM ON A CELLULOSE BASIS WHICH AN INSERT REINFORCES (AS AMENDED)

SECOND PRELIMINARY AMENDMENT

Director of Patents
Washington, D.C. 20231

Sir:

Prior to examination of the above-identified application, Applicants respectfully request that the following amendments be entered into the application:

IN THE ABSTRACT:

Page 18, please amend as follows:

The invention relates to a film tube (10) based on cellulose which is produced by extruding an aqueous cellulose-N-methyl-morpholine-N-oxide (NMMO) solution through a ring nozzle (21) onto a lining (3). The film tube (10) is manufactured by means of a vertically descending spinning in a spinning vat (12) in which a spinning bath (11) is located. The film tube (10) which is submerged in the spinning bath (11) passes through an air gap (9) between the underside of a nozzle block (7) and the upper surface of the spinning bath (11) and, internally, is pressurized, supported and slightly stretched in a transversal manner by compressed air. The film tube (10) is filled with an inner bath solution (31) via a supply tube (18). An idle roller (13) is

situated near the bottom of the spinning vat (12) in order to guide the film tube (10) around and, afterwards, out of the spinning vat (12) in an upward sloping manner. The film tube is laid flat along a contact section (27) of the idle roller (13).

REMARKS

Applicants respectfully request that the foregoing amendments be made prior to examination of the present application.

Respectfully submitted,

June 15, 2001
Date

Susan E. Shaw McBee

Susan E. Shaw McBee
Reg. No. 39,294

HELLER EHRMAN WHITE & McAULIFFE LLP
1666 K Street, N.W.
Suite 300
Washington, D.C. 20006
Tel: 202-912-2000
Fax: 202-912-2020

26633
PATENT TRADEMARK OFFICE

Application No. 09/787,416

MARKED-UP COPY OF AMENDED ABSTRACT

The invention relates to a film tube (10) based on cellulose which is produced by extruding an aqueous cellulose-N-methyl-morpholine-N-oxide (NMMO) solution through a ring nozzle (21) onto a lining (3). The film tube (10) is manufactured by means of a vertically descending spinning in a spinning vat (12) in which a spinning bath (11) is located. The film tube (10) which is submerged in the spinning bath (11) passes through an air gap (9) between the underside of a nozzle block (7) and the upper surface of the spinning bath (11) and, internally, is pressurized, supported and slightly [strcteched] stretched in a transversal manner by compressed air. The film tube (10) is filled with an inner bath solution (31) via a supply tube (18). An idle roller (13) is situated near the bottom of the spinning vat (12) in order to guide the film tube (10) around and, afterwards, out of the spinning vat (12) in an upward sloping manner. The film tube is laid flat along a contact section (27) of the idle roller (13).

09/787416
JC03 Rec'd PCT/PTO 22 MAR 2001

SUBSTITUTE SPECIFICATION

09787416.062501

09/787416
JC03 Rec'd PCT/PTO 22 MAR 2001

Method and Apparatus for the Manufacture of a Tube Made of Film on a Cellulose Basis Which an Insert Reinforces

BACKGROUND OF THE INVENTION

Field of the Invention

The invention relates to a method and an apparatus for the manufacture of a tube made of film on a cellulose basis, which an insert reinforces, by extruding an aqueous solution of cellulose-N-methyl-morpholin-N-oxide (NMMO) onto the insert, which is drawn from a roll and formed to a tube with an overlapping longitudinal seam.

Description of Related Art

Cellulose is not soluble in common solvents and has neither a melting point nor a melting range and therefore cannot be worked as a thermoplastic. For this reason cellulose is normally converted chemically for the manufacture of casings for foods, such as sausage casings, this process involving a degradation of the cellulose, i.e., the average degree of polymerization of the cellulose is lower. Such methods are technically very complicated and accordingly expensive to practice.

Presently the viscose method is preferred in the extrusion of film tubes on a cellulose basis. The cellulose is reacted with caustic soda solution and then reacted with carbon disulfide. Thus, a cellulose xanthate solution is obtained, which is extruded through a spinning or ring nozzle into a so-called spin bath or coagulating bath. The cellulose is regenerated by means of additional coagulating baths and washing baths.

It has long been known that cellulose is soluble in oxides of tertiary amines, and that at present the most appropriate solvent for cellulose is N-methyl-morpholin-N-oxide (NMMO). The cellulose dissolves therein, without changing chemically. No breakdown of cellulose chains takes place. The preparation of appropriate spinning solutions is known (DD 218 104; DD 298 789; US-A 4,145,532, US-A 4,196,282, US-A 4,255,300).

Filaments can be made from the solutions by extrusion into a spin bath (DE-A 44 09 609; US-A 5,417,909). In WO 95/07811 (= CA 2,149,218) there is also disclosed a method for the preparation of cellulose tubular films by the aminoxide method. What is distinctive of this method is the cooling of the extruded film with a cooling gas immediately under the ring gap of the extrusion nozzle. According to EP A 662 283, the extruded tubular film is cooled from within by a liquid.

Recovery and purification of the NMMO are described in DD 274 435. Since the cellulose is not chemically converted in the process the apparatus cost is lower. In the aminoxide method no gaseous or aqueous waste products are produced, so that there are no problems with exhausts or waste water. It is therefore acquiring increasing importance.

In EP-A 0 686 712 the production of flexible cellulose fibers by the N-methylmorpholin-N-oxide (NMMO) is described. In it a cellulose solution in aqueous NMMO is forced through a spinneret, carried across an air gap into an aqueous coagulating bath containing NMMO and then washed, finish-treated and dried.

According to WO 93/13670 a seamless, tubular food casing is made by extruding a solution of cellulose in NMMO/water by means of a special extrusion die. Between the extrusion die and the coagulating bath there is an air gap. Distinctive of this method is an especially formed hollow mandrel through which the coagulating liquid can circulate also inside of the tube. In the air gap the interior of the extruded tube is filled virtually completely with the hollow mandrel and the coagulating liquid. The tube is not stretched transversely.

In WO 95/35340 a method is described for the production of cellulose blowing films in which a non-derivatized cellulose dissolved in NMMO is used.

Document GB-A 1,042,182 describes a method for the production of a film tube on a cellulose basis reinforced by an insert by extruding a cellulose-NMMO solution onto the insert, the insert being drawn from a roll. The insert is formed into a tube with an overlapping longitudinal seam, the seam not being cemented. This tube is treated inside and out with a viscose solution.

Document DE-A 1 952 464 describes an apparatus for coating and imbibing a paper tube with polyvinyl alcohol resin. The paper tube is formed from a paper web. A cementing apparatus for cementing the overlapping longitudinal seam is not provided. Neither is any preheating of the paper web before it is coated with the polyvinyl alcohol resin performed.

In the document WO-A 95/07811 an apparatus is described for the production of tubes by extruding a cellulose-NMMO solution. The tubes are pure cellulose casings, but not so-called fiber casings in which a fiber insert strengthens the tube. The apparatus for the production of the tube is designed so that the tube can be cooled inside and out with air, so that the NMMO solution solidifies rapidly. The cooling air is by no means supporting air that is introduced into the interior of the film tube.

In the state of the art, methods and apparatus are known for the production of a fiber-reinforced cellulose tube by the viscose process, but these methods and apparatus are not applicable in NMMO technology for the following reasons:

- Different temperatures of the spinning solutions

- Different viscosities of the spinning solutions

- Different solvents

- Different sensitivities to evaporation and dilution, temperature fluctuations, and different temperature limits.

Usually the cellulose in the viscose process is spun in the temperature range of 20 to 45°C. Instead, the extrusion temperature of cellulose NMMO solutions is around 85 to 115°C,

The viscosity of cellulose in the viscose process is about 10 to 30 Pas, and that of NMMO solutions 10 to 300 Pas, especially 20 to 200 Pas. Cellulose in the viscose process

reacts with caustic soda solution and then with carbon disulfide, while the NMMO solutions are organic solutions.

SUMMARY OF THE INVENTION

The invention is therefore addressed to the problem of designing a method and an apparatus so that they will be suitable for coating inserts formed into a tube with cellulose-NMMO solutions and permit a uniform penetration of the inserts with cellulose-NMMO solutions.

This problem is solved by the invention in that the tube passes through a heating section situated ahead of the nozzle block and in communication therewith, in which the insert is preheated with hot air to the temperature of the extruded cellulose-NMMO solution, then the seam is cemented with straight NMMO or cellulose-NMMO solution and the tube is then carried through the nozzle block in which the cellulose-NMMO solution is applied to the tube and penetrates it to obtain an insert-reinforced film tube, that the interior of the film tube is filled with an aqueous NMMO solution, and that the film tube exits the nozzle block and enters a spin bath, is turned around in the latter, and is carried out of it.

DETAILED DESCRIPTION OF A PREFERRED EMBODIMENT

In an embodiment of the process, after it is drawn from the roll, emulsifiers, wetting agents and/or anchoring agents are applied by one of the known methods such as roller application. An appropriately pressure-controlled supporting air is blown into the interior of the film tube after it leaves the nozzle block.

As the process continues, the film tube is carried through a heated annular gauging disk through which a heating medium flows in a controlled circuit.

In embodiment of the process, aqueous NMMO solution is delivered into the interior of the film tube and also removed therefrom, the delivery and removal being performed at a distance apart from one another. At the same time the level of delivery of the aqueous

NMMO solution in the film tube is adjustable and its removal is performed such that the level in the film tube is variably up to 20 mm higher and up to 45 mm lower than the level in the spin bath.

The rest of the process is to be found in the features of claims 7 to 12.

As a variant of this process it is also possible, instead of passing through a tub filled with the spin bath, to apply the spin bath directly internally and externally onto the film tube, through ring nozzles for example, as is described in EP-A0 006 601. The spin bath level is then lowered inside and out to the top edge of the spin tub's deflector roll.

The apparatus for producing a film tube on a cellulose basis, which the insert reinforces, by extruding an aqueous cellulose-N-methyl-morpholin-N-oxide-(NMMO) solution onto the insert, with a nozzle block and a spin bath, is characterized in that a supply roll for the insert, a deflector roll, and a forming section in which the insert is formed into a tube with an overlapping longitudinal seam, are present, that a preheating system for the tube is disposed ahead of the nozzle block, that the preheating system is connected by hot air ducts and an exhaust duct is connected with a controllable heater out of which air heated in the circuit flows into the preheating system, and from which cooled air flows back into the heater, and that the tube runs through the nozzle block which is preceded by a cementing system for cementing the longitudinal seam of the tube and which contains a ring nozzle out of the nozzle gap of which the cellulose-NMMO solution is applied to the tube preheated to the temperature of the extrusion solution in order to complete the formation of the film tube.

In further embodiment of the apparatus, the insert is selected from the group, paper, nonwoven, fiber fleece, fiber paper, the fibers being especially long hemp fibers.

In embodiment of the apparatus, after the insert is drawn from the supply roll an applicator system is attached, with which additives such as emulsifiers, wetting and/or anchoring agents can be applied to the insert and can be dried in the following hot open air section.

It is also possible that the preheating system is not required in every case, so that in certain manufacturing procedures it remains shut off.

In an embodiment of the invention, the nozzle block contains a ring nozzle which is heated by a heating medium, and a delivery tube and a removal tube for the aqueous NMMO solution, plus a duct for supporting air for the film tube, are brought centrally through an annular gauging disk which is arranged concentrically with the ring nozzle in the interior of the film tube and forms with the latter an annular gap through which the film tube passes.

The annular gauging disk is connected with the heating circuit for heating.

In embodiment of the apparatus, the infeed tube and the outfeed tube are adjustable for height within the film tube.

The further configuration of the apparatus will be apparent from the features of claims 19 to 25.

By the method of the invention a substantially uniform penetration of the insert with the cellulose-NMMO solution, so that, after passing through additional treatment steps, such as precipitation or coagulation baths, a composite of a fiber-reinforced film tube is obtained which has improved properties for its use. The fiber-reinforced film tube on a cellulose-NMMO basis corresponds in its properties to the known cellulose fiber or fiber casings which are made from cellulose hydrate and reinforced with wet-strengthened fibers of cellulose (= cellulose fiber fleece).

The invention is further explained below with the aid of the drawings, wherein:

Fig. 1 is a schematic sectional view of the apparatus according to the invention with a height-adjusted infeed tube in the film tube.

Fig. 2 is a schematic sectional view of an apparatus similar to that of Fig. 1, with infeed tube lowered into the film tube.

Fig. 3 is an enlarged sectional view at point A in Fig. 1, and

Figs. 4a and 4b are side and top views of a cementing device for a tubular envelope, formed from an insert.

An apparatus 1 shown in Fig. 1 for extruding an aqueous cellulose-N-methyl-morpholin-N-oxide (NMMO) solution onto an insert comprises a supply roll 2 for the insert 3, a deflector roll 4, a shaping section 5, a nozzle block 7 with a ring nozzle and a spin tub 12 which is filled with a spinning or precipitation bath 11.

The insert 3, which is paper, nonwoven, fiber paper or fiber fleece wherein the fibers are preferably hemp fibers, is drawn from the supply roll 2 and carried over the deflector roll 4. The fiber paper and the fiber fleece are solidified wet when manufactured, by being impregnated with dilute viscose, cellulose acetate solution or plastic washes. In these embodiments the insert 3 is used with preference. Ahead of the deflector roll 4 is an applicator 39 comprising a grid cylinder 40, a squeegee 33 and a pair of pinch rolls 34-35 for the application of additives, such as emulsifiers, wetting agents or sticking agents, to the insert 3. After the insert 3 passes over the deflector roll 4 the formation of a tube 6 with an overlapping longitudinal seam 38 (see Fig. 4a) takes place in the shaping section 5 by means of a forming shoulder not shown.

The tube 6 and the film tube 10 which it forms is made by a vertically descending spinning. For this, the tube 6 passes through the ring nozzle 21 in the nozzle block 7 through the gap of which the cellulose-NMMO solution is extruded onto the envelope 6 to complete the formation of the film tube 10. First the longitudinal seam on the envelope 6 is cemented ahead of the nozzle block 7 by a cementing system 25 shown more in detail in Figures 4a and 4b; straight NMMO or cellulose-NMMO solutions serve as the adhesive at temperatures between 15 and 110°C, especially the temperature of the cellulose-NMMO solution that is to be applied.

The extruded cellulose-NMMO solution coats and penetrates uniformly through the tubular envelope 6. The pressure required for penetration is built up by the geometry of the annular gap 26 in the nozzle body 7, which amounts to 0.1 to 5 mm, especially 0.5 to 1.5 mm.

The annular gap 26 is formed by a gauging disk 8 and the inside of the annular nozzle 21.

After exiting from the nozzle body 7 the film tube 10 passes through an air section 9 before it plunges into the spin bath 11 in the spin tub 12. In the air section 9 a temperature treatment with temperature-controlled air can take place if necessary, in which case heated air delays the solidification of the cellulose-NMMO solution and cool air accelerates it.

Instead of the one-sided application of the cellulose-NMMO solution to the outside of the envelop 6, the cellulose-NMMO solution can also be applied bilaterally, i.e., both to the outside and to the inside of the envelope 6.

The ring nozzle 21 serves as the outer ring nozzle, while the inner ring nozzle takes the place of the gauging disk 8 in Figures 1 and 2. The inner ring nozzle is, like the gauging ring disk 8, heatable. In this variant of the treatment, the outer and inner cellulose-NMMO solution filmcan be applied spaced apart from one another, i.e., delayed in time.

The spin bath 11 consists of an aqueous NMMO solution with an NMMO content 5 to 50 weight-percent, especially 8 to 20 weight-percent. A preheating system 15 is arranged in front of the nozzle block 7 and connected to a controlled heater 17 by hot air ducts 22 and 23 and an exhaust duct 24. The aqueous spinning solution extruded from the ring nozzle 21 is a cellulose-NMMO solution with a morpholine content of 75 to 90 wt.-%, especially 87.7 wt.-%. The spinning solution is fed on one side into the ring nozzle 21 by means of a spinning pump, not shown, and distributed in a largely uniform manner over the circumference through a nozzle gap through a distributor plate, not shown. The ring nozzle 21 has a double jacket 32 for heating the ring nozzle 21 to the temperature of the morpholine solution, a heating medium being provided for the purpose, which flows through the double jacket 32 and is heated in a controlled heating circuit 16 which is connected by lines to the double jacket 32. The film tube 10 extruded from the nozzle block 7 passes through the air section 9 in which it is expanded by compressed air and stretched slightly crosswise. The expanded film tube 10 has no contact with the outside of a tube 29 which extends downward past the bottom of the nozzle block 7. The air section amounts to 1 to 1000 mm, especially 200 to 500 mm. The tube 29 surrounds an inlet

and outlet tube 18 and 19, respectively, for an internal bath solution 31 which fills the film tube 10. This internal bath solution is an aqueous NMMO solution with an NMMO content of 5 to 50 wt.-%, especially 8 to 20 wt.-%. The inlet and outlet tubes 18 and 19 extend vertically downward into the film tube 10 plunging into the spin bath 11. As the feeding of the internal bath solution 31 into the film tube 10 begins, the inlet tube 18 assumes a raised position, as shown in Fig. 1. As soon as the film tube is filled with the internal bath solution, the inlet tube 18 is advanced to a position in the film tube 10 which is just above the deflector roll 13 for the film tube 10, as can be seen in Fig. 2. The inlet tube 18 can be raised and lowered within the vertically plunging film tube 10, and so can the outlet tube 19.

Air at a pressure of 0.1 to 10 mbar above atmospheric is passed through a line 20 for supporting air into the interior of the film tube 10. This pressure produces a slight expansion of the inflated film tube in the air section 9 directly following its exit from the nozzle block 7. The film tube 10 plunging into the spin bath 11 is turned around near the bottom of the spin tube 12. A deflector roll 13 is provided, which is power-driven, and around which the film tube 10 is passed. After the film tube turns around it is brought up through the spin bath 11 and out of the bath at an angle. The film tube 10 running upward at an angle is pinched together by the spin bath pressure just underneath the surface of the spin bath and is driven in the flattened state out of the spin bath 11. Wipers 14 on both sides of the collapsed film tube 10 wipe away the excess spin bath solution. The width of the flattened film tube 10 is kept as constant as possible. Any departure of the width of the flattened film tube 10 from the desired dimension results in a readjustment of the tension applied to the film tube in order to maintain the specified dimension.

Fig. 2 differs from Fig.1 only in that the inlet tube 18 is inserted so far down into the vertically descending film tube 10, in comparison to Fig. 1, that the mouth of the inlet tube is situated just above the deflector roll 13. The spin bath 11 and the internal bath solution31 are, as mentioned previously, aqueous NMMO solutions which, as the extrusion of the film tube 10 begins, have the same NMMO concentration. As extrusion progresses, the NMMO concentration of the inner bath solution 31 will increase, since morpholine penetrates the insert 3 during the cellulose regeneration, and enters into the internal bath solution 31 and

concentrates therein. Since morpholine has a greater density than water, the concentration or density of the NMMO solution increases toward the deflector roll 13 inside of the film tube 10. The concentration of the NMMO solution in the spin bath 31 varies hardly at all, since the morpholine yielded by the film tube to the spin bath 11 can increase the NMMO concentration of the spin bath 11 to only a negligible degree. In the inside bath solution 31 in the film tube 10, unless the NMMO concentration in the inside bath solution 31 is regulated, different coagulation conditions might occur, as well as a variation of the diameter of the film tube 10. Due to the constant delivery and removal of the inside bath solution 31 through the delivery and removal tube 18 and 19, respectively, a constant renewal of the inside bath solution 31 occurs, i.e., the inside bath solution 31 enriched with morpholine near the deflector roll 13 is diluted, so that the NMMO concentration of the inside bath solution 31 near the deflector roll 13 is less than or at most equal to the NMMO concentration of the spin bath 11. The tension applied to the film tube 10, together with thepressure of the spin bath 11, is enough to urge the film tube 10 against the deflector roll 13 along the line of contact 27 so that it is more or less flattened, as can be seen in Figures 1 and 2. Thus, uniform pressure conditions are established in the film tube over the entire length from just beneath the surface of the spin bath 11 to an area close to the deflector roll 13, so that the gauge or diameter of the film tube 10 is constant and shows no fluctuation or narrowing. The density of the inside bath solution 31 is thus dependent upon the throughput of the inside bath solution or morpholine solution, the amount of the inside bath and the depth of immersion of the inlet tube 18 or the point at which fresh morpholine solution is fed into the inside bath solution. The position or point at which the inside bath solution 31 flows into the film tube 10 substantially influences the gauge constancy, the level of the inside bath in the ascending film tube 10 after the deflector roll 13 and the location for the removal of the inside bath solution 31 from the interior of the film tube.

Fig. 3 shows on an enlarged scale the section indicated at A in Fig. 1. The tube 29 is lowered about 50 to 100 mm into the inside bath solution in the film tube. The aspirating tube 19 is in a position in which it establishes a level 30 inside of the tube 29, which is up to 20 mm higher or 45 mm lower than an inside bath level 28 of the inside bath solution of the film tube 10.

In other words, this means that the aspirating tube 19 assumes a position at which the inside bath solution is drawn up to a distance of as much as 20 mm higher or 45 mm lower than the tube level 28. The highest and the lowest tube levels 30 are indicated by broken lines 30_{max} and 30_{min} in Fig. 3. The aspiration usually starts below the level of the spin bath 11, so that the air section 9 situated above it and the pressure conditions there prevailing have no influence on the inside bath solution and thus cannot produce any gauge fluctuations in the film tube 10, either. If the aspiration is performed above the level of the spin bath 11, the effect of the pressure conditions in the air section 9 on the film tube 10 is negligible, since the latter is made stable in shape by the insert to the extent that it is hardly subject to any gauge variations.

Due to the adjustment of the depth of immersion of the inlet tube 18 and the constant renewal of the inside bath solution the density of the inside bath solution 31 is kept at a uniform level, which results in a constriction of the film tube 10 along the line of contact 27 with the deflector roll 13 and keeps the level of the inside bath solution 31 in the ascending film tube 10 constant for as long as desired with respect to the surface of the spin bath 11, so that irregular running and film tube gauge variations no longer occurs. The constant renewal and the minimum delivery of inside bath solution 31 are to be determined individually for each rate of extrusion or output of the film tube.

The film tube 10 emerging from the spin bath 11 then passes through precipitation and washing tubs not shown, and can also be treated with plasticizers, for example, and then dried before being wound up and further treated.

In a variation of the method, the level of the spin bath 11 inside and outside of the film tube is lowered to the upper edge of the deflector roll 13 and the film tube 10 can be sprayed inside and out with the spin bath through ring nozzles, as described, for example, in EP-A 0 006 601.

In Figures 4a and 4b, a side view and top view of the cementing system 25 are shown schematically. The cementing system 25 comprises a hollow, fixed finger 36 with a slit 37 from which the cement flowing inside of the finger 36 is applied to the longitudinal seam 38 of the

tubular envelope 6. The envelope 6 is simultaneously moving vertically in the direction of the arrow to the finger 36.

The film tube 10 can be expanded alternatively or additionally to the supporting air through a tube, a ring or a spreader in flat form, free of wrinkles.

Besides the treatment of the film tube 10 in the spin bath 11, as described in Figures 1 to 4b, with complete immersion of the film tube in the spin bath, wherein the tube interior is filled with an inside bath for pressure equalization, and the inside bath level 28 can be regulated differently from the tube level 30 in the tube 29 or by the outside level of the spin bath 11, it is also possible, in the case of the previously described bilateral coating with cellulose-NMMO solution, a film of NMMO solution can be applied to the outside as well as to the inside of the film tube 10 by means of an outer and inner annular ring nozzle.

What is Claimed Is:

1. Method for the production of a film tube on a cellulose basis, which is strengthened by an insert, by extruding an aqueous cellulose-N-methyl-morpholine N-oxide (NMMO) solution onto the insert, which is drawn from a roll and formed into a tube with overlapping longitudinal seam, characterized in that the tube passes through a heating section situated ahead of the nozzle block and communicating therewith, in which the insert is preheated with hot air to the temperature of the extruded cellulose-NMMO solution, and then the seam is cemented with pure NMMO or cellulose-NMMO solution, and the tube is then carried through the nozzle block in which the cellulose-NMMO solution is applied to the tube and penetrates it, in order to obtain an insert-reinforced film tube, that the interior of the film tube is filled with an aqueous NMMO solution, and that the film tube exits from the nozzle block and enters into a spin bath, is turned about in the latter and is carried out.

2. Method according to claim 1, characterized in that emulsifiers, wetting agents and/or anchoring agents are applied by one of the known methods such as roller application.

3. Method according to claim 1, characterized in that pressure-regulated supporting air is blown into the interior of the film tube after departure from the nozzle block.

4. Method according to claim 1, characterized in that the film tube is carried through a heated annular gauging disk through which a heating medium flows in a controlled circuit.

5. Method according to claim 1, characterized in that the aqueous NMMO solution is delivered through the nozzle block into the interior of the film tube and also removed from it, the delivery and removal being performed at a distance apart from one another.

6. Method according to claim 5, characterized in that the level of the delivery of the aqueous NMMO solution is adjustable and that the removal is performed such that the level in the film tube is variably higher by up to 20 mm and lower by up to 45 mm than the level in the spin bath.

7. Method according to claim 1, characterized in that the film tube, after leaving the nozzle block, runs through an air section until it enters into the spin bath, and that in the air section an external temperature treatment takes place which regulates the rate of solidification of the cellulose-NMMO solution of the film tube.

8. Method according to claim 1, characterized in that the film tube plunges vertically into the spin bath and with maintenance of a constant tension is turned about by a powered return roll running close to the bottom of the spin bath tube and is carried out upwardly at an angle from the spin bath.

9. Method according to claim 1, characterized in that the spin bath level inside and outside of the film tube is lowered as far as the upper edge of a return roll and that the film tube is sprayed inside and out with spin bath.

10. Method according to claim 1, characterized in that the longitudinal seam of the tubular envelope is cemented with straight NMMO or a cellulose-NMMO solution at a temperature of 15 to 110°C, especially at the temperature of the cellulose-NMMO solution extruded in the nozzle block.

11. Method according to claim 1, characterized in that the cellulose content of the extruded cellulose-NMMO solution amounts to 1 to 15 wt.%, especially 3 to 7 wt.-% with respect to the total solution, and that the average degree of polymerization ranges from 250 to 800, especially from 300 to 500.

12. Method according to claim 1, characterized in that the aqueous NMMO solution of the spin bath has an NMMO concentration of 5 - 50 wt.-%, especially of 8 to 20 wt.-% and that the spin bath is adjusted to 0 to 50°C, especially 2 to 20°C.

13. Apparatus for producing a film tube on a cellulose basis, which an insert reinforces, by extruding an aqueous cellulose-N-methylmorpholin-N-oxide (NMMO) solution onto the insert, with a nozzle block (7) and a spin bath (11), characterized in that a supply roll (2) for the insert (3), a deflector roll (4), and a forming section (5) in which the insert (3) is formed into a tube (6) with overlapping longitudinal seam, are present, that a preheating system (15) for the tube (6) is disposed ahead of the nozzle block (7), that the preheating system (15) is connected by hot air ducts (22, 23) and an exhaust duct (24) to a controllable heater (17) from which air heated in the circuit flows into the preheating system (15) and from which cooled air flows back into the heater (17), and that the tube (6) passes through the nozzle block (7) which is preceded by a cementing system (25) for cementing the longitudinal seam and which contains an annular nozzle (21) from whose nozzle gap the cellulose-NMMO solution is applied to the tube (6) preheated to the temperature of the extrusion solution for the formation of the film tube (10).

14. Apparatus for the production of a film tube according to claim 13, characterized in that the insert (3) is selected from the group, paper, nonwoven, fiber fleece, fiber paper, the fibers being especially long hemp fibers.

15. Apparatus for the production of a film tube according to claim 13, characterized in that, after the drawing of the insert (3) from the supply roll (2) an applicator system (39) is provided by which additives, such as emulsifiers, wetting agents and/or anchoring means can be applied

to the insert and dried in the following hot open air section.

16. Apparatus for the production of a film tube according to claim 13, characterized in that the nozzle block (7) contains a ring nozzle (21) which is heated by a heating medium, and that the delivery tube, the removal tube (18, 19) and the duct (20) for the air supporting the film tube (10) are brought centrally through a gauging ring disk (8) which is arranged concentrically with the ring nozzle (21) in the film tube interior and forms with the latter an annular gap (26) through which the film tube (10) runs.

17. Apparatus for the production of a film tube according to claim 16, characterized in that the gauging ring disk (8) is connected to the heating circuit (16) for the purpose of heating.

18. Apparatus for the production of a film tube according to claim 16, characterized in that the delivery tube (18) and the removal tube (19) are individually height-adjustable within the film tube (10).

19. Apparatus for the production of a film tube according to claim 18, characterized in that the delivery tube (18) is disposed in an upper position at the beginning of the delivery of the aqueous NMMO solution into the film tube (10) and at the start of continuous operation assumes a position above the return roll (13).

20. Apparatus for the production of a film tube according to claim 16, characterized in that the heating medium flows through the ring nozzle and is carried in a controlled heating circuit (16).

21. Apparatus for the production of a film tube according to claim 13, characterized in that the air section (9) amounts to 1 to 1000 mm, especially 200 to 500 mm, and that if necessary the film tube (10) can be heated to delay its solidification or cooled to accelerate its solidification in the air section.

22. Apparatus for the production of a film tube according to claim 13, characterized in that the

return roll (13) disposed near the bottom of the spin tub (12) is driven and exerts a constant tension on the vertically descending film tube (10).

23. Apparatus for the production of a film tube according to claim 22, characterized in that the film tube (10) lies flat against the return foll (13) along a line of contact (27) as a result of the tension exerted on the film tube (10).

24. Apparatus for the production of a film tube according to claim 13, characterized in that the spin bath (11) and the aqueous NMMO solution in the film tube (10) have equal NMMO concentrations at the beginning of the extrusion of the film tube (10).

25. Apparatus for the production of a film tube according to claim 13, characterized in that the excess pressure of the supporting air in the film tube (10) amounts to 0.1 to 10 mbar in the range of the air section (9).

ABSTRACT OF THE DISCLOSURE

The invention relates to a film tube (10) based on cellulose which is produced by extruding an aqueous cellulose-N-methyl-morpholine-N-oxide (NMMO) solution through a ring nozzle (21) onto a lining (3). The film tube (10) is manufactured by means of a vertically descending spinning in a spinning vat (12) in which a spinning bath (11) is located. The film tube (10) which is submerged in the spinning bath (11) passes through an air gap (9) between the underside of a nozzle block (7) and the upper surface of the spinning bath (11) and, internally, is pressurized, supported and slightly strcteched in a transversal manner by compressed air. The film tube (10) is filled with an inner bath solution (31) via a supply tube (18). An idle roller (13) is situated near the bottom of the spinning vat (12) in order to guide the film tube (10) around and, afterwards, out of the spinning vat (12) in an upward sloping manner. The film tube is laid flat along a contact section (27) of the idle roller (13).

Method and Apparatus for the Manufacture of a Tube Made of Film on a Cellulose Basis Which Reinforces an Insert

The invention relates to a method and an apparatus for the manufacture of a tube made of film on a cellulose basis, which reinforces an insert, by extruding an aqueous solution of cellulose-N-methyl-morpholin-N-oxide (NMMO) onto the insert.

Cellulose is not soluble in common solvents and has neither a melting point nor a melting range and therefore cannot be worked as a thermoplastic. For this reason cellulose is normally converted chemically for the manufacture of casings for foods, such as sausage casings, this process involving a degradation of the cellulose, i.e., the average degree of polymerization of the cellulose is lower. Such methods are technically very complicated and accordingly expensive to practice.

Presently the viscose method is preferred in the extrusion of film tubes on a cellulose basis. The cellulose is reacted with caustic soda solution and then reacted with carbon disulfide. Thus, a cellulose xanthate solution is obtained, which is extruded through a spinning or ring nozzle into a so-called spin bath or coagulating bath. The cellulose is regenerated by means of additional coagulating baths and washing baths.

It has long been known that cellulose is soluble in oxides of tertiary amines, and that at present the most appropriate solvent for cellulose is N-methyl-morpholin-N-oxide (NMMO). The cellulose dissolves therein, without changing chemically. No breakdown of cellulose chains takes place. The preparation of appropriate spinning solutions is known (DD 218 104; DD 298 789; US-A 4,145,532, US-A 4,196,282, US-A 4,255,300).

Filaments can be made from the solutions by extrusion into a spin bath (DE-A 44 09 609; US-A 5,417,909). In WO 95/07811 (= CA 2,149,218) there is also

disclosed a method for the preparation of cellulose tubular films by the aminoxide method. What is distinctive of this method is the cooling of the extruded film with a cooling gas immediately under the ring gap of the extrusion nozzle. According to EP A 662 283, the extruded tubular film is cooled from within by a liquid.

Recovery and purification of the NMMO are described in DD 274 435. Since the cellulose is not chemically converted in the process the apparatus cost is lower. In the aminoxide method no gaseous or aqueous waste products are produced, so that there are no problems with exhausts or waste water. It is therefore acquiring increasing importance.

In EP-A 0 686 712 the production of flexible cellulose fibers by the N-methylmorpholin-N-oxide (NMMO) is described. In it a cellulose solution in aqueous NMMO is forced through a spinneret, carried across an air gap into an aqueous coagulating bath containing NMMO and then washed, finish-treated and dried.

According to WO 93/13670 a seamless, tubular food casing is made by extruding a solution of cellulose in NMMO/water by means of a special extrusion die. Between the extrusion die and the coagulating bath there is an air gap. Distinctive of this method is an especially formed hollow mandrel through which the coagulating liquid can circulate also inside of the tube. In the air gap the interior of the extruded tube is filled virtually completely with the hollow mandrel and the coagulating liquid. The tube is not stretched transversely.

In WO 95/35340 a method is described for the production of cellulose blowing films in which a non-derivatized cellulose dissolved in NMMO is used.

In the state of the art, methods and apparatus are known for the production of a fiber-reinforced cellulose tube by the viscose process, but these methods and apparatus are not applicable in NMMO technology for the following reasons:

- Different temperatures of the spinning solutions
- Different viscosities of the spinning solutions
- Different solvents
- Different sensitivities to evaporation and dilution, temperature fluctuations, and different temperature limits.

Usually the cellulose in the viscose process is spun in the temperature range of 20 to 45°C. Instead, the extrusion temperature of cellulose NMMO solutions is around 85 to 115°C,

The viscosity of cellulose in the viscose process is about 10 to 30 Pas, and that of NMMO solutions 10 to 300 Pas, especially 20 to 200 Pas. Cellulose in the viscose process reacts with caustic soda solution and then with carbon disulfide, while the NMMO solutions are organic solutions.

The invention is therefore addressed to the problem of designing a method and an apparatus so that they are suitable for coating inserts formed into a tube with cellulose-NMMO solutions and permit a uniform penetration of the inserts with cellulose-NMMO solutions.

This problem is solved as regards process in that the insert is drawn from a supply roll, treated with emulsifiers, wetting agents and/or anchoring agents and formed into a tubular envelope with an overlapping longitudinal seam which is cemented ahead of a nozzle block through which the envelope is carried and in which the cellulose-NMMO solution is applied to the envelope and

penetrates it in order to obtain an insert-reinforced film tube, that the interior of the film tube is filled with an aqueous NMMO solution, and that the film tube exits from the nozzle block and enters into a spinning bath, turned about in the latter and carried out of it.

In embodiment of the process, the tubular envelope passes through a heating section ahead of the nozzle block, in which it is preheated with hot air to the temperature of the extruded cellulose-NMMO solution. A controlled-pressure supporting air is blown into the interior of the film tube after it leaves the nozzle block

As the process continues, the film tube is carried through a heated annular gauging disk through which a heating medium flows in a controlled circuit.

In embodiment of the process, aqueous NMMO solution is delivered into the interior of the film tube and also removed therefrom, the delivery and removal being performed at a distance apart from one another. At the same time the level of delivery of the aqueous NMMO solution in the film tube is adjustable and its removal is performed such that the level in the film tube is variably up to 20 mm higher and up to 45 mm lower than the level in the spin bath.

The rest of the process is to be found in the features of claims 7 to 12.

As a variant of this process it is also possible, instead of passing through a tub filled with the spin bath, to apply the spin bath directly internally and externally onto the film tube, through ring nozzles for example, as is described in EP-A

0 006 601. The spin bath level is then lowered inside and out to the top edge of the spin tub pulley.

The apparatus for the production of a film tube on a cellulose basis, which the insert reinforces, by extruding an aqueous cellulose-N-methyl-morpholin-N-oxide-(NMMO) solution onto the insert, with a nozzle block and a spin bath, is characterized in that a supply roll for the insert, a deflector roll with a device for applying additives to the insert carried from the supply roll over the deflector roll, a forming section in which the insert is shaped to a tubular envelope with overlapping longitudinal seam are present, that the tubular envelope passes through the nozzle block which is preceded by a cementing device for cementing the longitudinal seam of the tubular envelope and which contains a ring nozzle from the nozzle gap of which the cellulose-NMMO solution is extruded onto the tubular envelope to form a film tube, that between the exit from the nozzle block and the spin bath a temperature-controlled air section is present in a spin tub, that near the bottom of the spin tub a return roll for the film tube plunging vertically into the spin bath is disposed, and that a delivery and removal tube for the aqueous NMMO solution as well as a duct for supporting air are situated in the interior of the film tube.

In further embodiment of the invention the insert is selected from the group, paper, nonwoven, fiber mat and fiber paper, wherein the fibers are especially long hemp fibers. In addition, a system for preheating the tubular envelope is disposed ahead of the nozzle block and the preheating system is connected by hot air ducts and an exhaust line to a controllable heater out of which air heated in the circuit flows into the preheating system and from which cooled air flows back into the heater. It is also possible that the preheating system is not needed in every case, so that it remains shut off in certain production procedures. It is also conceivable that the apparatus according to the invention can be operated without any such preheating system.

In an embodiment of the invention, the nozzle block contains a ring nozzle which is heated by a heating medium and the infeed and outfeed tube and the duct for the air supporting the film tube are brought centrally through an annular calibration disk which is arranged concentrically with the ring nozzle in the interior of the film tube and forms with the latter an annular gap through which the film tube passes.

The annular calibration disk is connected with the heating circuit for heating.

In embodiment of the apparatus, the infeed tube and the outfeed tube are adjustable for height within the film tube.

The further configuration of the apparatus will be apparent from the features of claims 19 to 25.

By the method of the invention a substantially uniform penetration of the insert with the cellulose-NMMO solution, so that, after passing through additional treatment steps, such as precipitation or coagulation baths, a composite of a fiber-reinforced film tube is obtained which has improved properties for its use. The fiber-reinforced film tube on a cellulose-NMMO basis corresponds in its properties to the known cellulose fiber or fiber casings which are made from cellulose hydrate and reinforced with wet-strengthened fibers of cellulose (= cellulose fiber fleece).

The invention is further explained below with the aid of the drawings, wherein:

Fig. 1 is a schematic sectional view of the apparatus according to the invention with a height-adjusted infeed tube in the film tube.

Fig. 2 is a schematic sectional view of an apparatus similar to that of Fig. 1, with infeed tube lowered into the film tube;

Fig. 3 is an enlarged sectional view at point A in Fig. 1, and

Figs. 4a and 4b are side and top views of a cementing device for a tubular envelope, formed from an insert.

An apparatus 1 shown in Fig. 1 for extruding an aqueous cellulose-N-methyl-morpholin-N-oxide (NMMO) solution onto an insert comprises a supply roll 2 for the insert 3, a deflector roll 4, a shaping section 5, a nozzle block 7 with a ring nozzle and a spin tub 12 which is filled with a spinning or precipitation bath 11.

The insert 3, which is paper, nonwoven, fiber paper or fiber fleece wherein the fibers are preferably hemp fibers, is drawn from the supply roll 2 and carried over the deflector roll 4. The fiber paper and the fiber fleece are solidified wet when manufactured, by being impregnated with dilute viscose, cellulose acetate solution or plastic washes. In these embodiments the insert 3 is used with preference. Ahead of the deflector roll 4 is an applicator 31 comprising a grid cylinder 32, a squeegee 33 and a pair of pinch rolls 34-35 for the application of additives, such as emulsifiers, wetting agents or sticking agents, to the insert 3. After the insert 3 passes over the deflector roll 4 the formation of a tubular envelope 6 with an overlapping longitudinal seam 38 (see Fig. 4a) takes place in the shaping section 5 by means of a forming shoulder not shown.

The tubular envelope 6 and the tube 10 which it forms is made by a vertically descending spinning. For this, the envelope 6 passes through the ring nozzle 21 in the nozzle block 7 through the gap of which the cellulose-NMMO

solution is extruded onto the envelope 6 to complete the formation of the film tube 10. First the longitudinal seam on the envelope 6 is cemented ahead of the nozzle block 7 by a cementing system 25 shown more in detail in Figures 4a and 4b; straight NMMO or cellulose-NMMO solutions serve as the adhesive at temperatures between 15 and 110°C, especially the temperature of the cellulose-NMMO solution that is to be applied.

The extruded cellulose-NMMO solution coats and penetrates uniformly through the tubular envelope 6. The pressure required for penetration is built up by the geometry of the annular gap 26 in the nozzle body 7, which amounts to 0.1 to 5 mm, especially 0.5 to 1.5 mm. The annular gap 26 is formed by a gauging disk 8 and the inside of the annular nozzle 21.

After exiting from the nozzle body 7 the film tube 10 passes through an air section 9 before it plunges into the spin bath 11 in the spin tub 12. In the air section 9 a temperature treatment with temperature-controlled air can take place if necessary, in which case heated air delays the solidification of the cellulose-NMMO solution and cool air accelerates it.

Instead of the one-sided application of the cellulose-NMMO solution to the outside of the envelop 6, the cellulose-NMMO solution can also be applied bilaterally, i.e., both to the outside and to the inside of the envelope 6.

The ring nozzle 21 serves as the outer ring nozzle, while the inner ring nozzle takes the place of the gauging disk 8 in Figures 1 and 2. The inner ring nozzle is, like the gauging ring disk 8, heatable. In this variant of the treatment, the outer and inner cellulose-NMMO solution filmcan be applied spaced apart from one another, i.e., delayed in time.

The spin bath 11 consists of an aqueous NMMO solution with an NMMO content 5 to 50 weight-percent, especially 8 to 20 weight-percent. A preheating system

15 is arranged in front of the nozzle block 7 and connected to a controlled heater 17 by hot air ducts 22 and 23 and an exhaust duct 24. The aqueous spinning solution extruded from the ring nozzle 21 is a cellulose-NMMO solution with a morpholine content of 75 to 90 wt.-%, especially 87.7 wt.-%. The spinning solution is fed on one side into the ring nozzle 21 by means of a spinning pump, not shown, and distributed in a largely uniform manner over the circumference through a nozzle gap through a distributor plate, not shown. The ring nozzle 21 has a double jacket 32 for heating the ring nozzle 21 to the temperature of the morpholine solution, a heating medium being provided for the purpose, which flows through the double jacket 32 and is heated in a controlled heating circuit 16 which is connected by lines to the double jacket 32. The film tube 10 extruded from the nozzle block 7 passes through the air section 9 in which it is expanded by compressed air and stretched slightly crosswise. The expanded film tube 10 has no contact with the outside of a tube 29 which extends downward past the bottom of the nozzle block 7. The air section amounts to 1 to 1000 mm, especially 200 to 500 mm. The tube 29 surrounds an inlet and outlet tube 18 and 19, respectively, for an internal bath solution 31 which fills the film tube 10. This internal bath solution is an aqueous NMMO solution with an NMMO content of 5 to 50 wt.-%, especially 8 to 20 wt.-%. The inlet and outlet tubes 18 and 19 extend vertically downward into the film tube 10 plunging into the spin bath 11. As the feeding of the internal bath solution 31 into the film tube 10 begins, the inlet tube 18 assumes a raised position, as shown in Fig. 1. As soon as the film tube is filled with the internal bath solution, the inlet tube 18 is advanced to a position in the film tube 10 which is just above the deflector roll 13 for the film tube 10, as can be seen in Fig. 2. The inlet tube 18 can be raised and lowered within the vertically plunging film tube 10, and so can the outlet tube 19.

Air at a pressure of 0.1 to 10 mbar above atmospheric is passed through a line 20 for supporting air into the interior of the film tube 10. This pressure produces a slight

expansion of the inflated film tube in the air section 9 directly following its exit from the nozzle block 7. The film tube 10 plunging into the spin bath 11 is turned around near the bottom of the spin tube 12. A deflector roll 13 is provided, which is power-driven, and around which the film tube 10 is passed. After the film tube turns around it is brought up through the spin bath 11 and out of the bath at an angle. The film tube 10 running upward at an angle is pinched together by the spin bath pressure just underneath the surface of the spin bath and is driven in the flattened state out of the spin bath 11. Wipers 14 on both sides of the collapsed film tube 10 wipe away the excess spin bath solution. The width of the flattened film tube 10 is kept as constant as possible. Any departure of the width of the flattened film tube 10 from the desired dimension results in a readjustment of the tension applied to the film tube in order to maintain the specified dimension.

Fig. 2 differs from Fig.1 only in that the inlet tube 18 is inserted so far down into the vertically descending film tube 10, in comparison to Fig. 1, that the mouth of the inlet tube is situated just above the deflector roll 13. The spin bath 11 and the internal bath solution31 are, as mentioned previously, aqueous NMMO solutions which, as the extrusion of the film tube 10 begins, have the same NMMO concentration. As extrusion progresses, the NMMO concentration of the inner bath solution 31 will increase, since morpholine penetrates the insert 3 during the cellulose regeneration, and enters into the internal bath solution 31 and concentrates therein. Since morpholine has a greater density than water, the concentration or density of the NMMO solution increases toward the deflector roll 13 inside of the film tube 10. The concentration of the NMMO solution in the spin bath 31 varies hardly at all, since the morpholine yielded by the film tube to the spin bath 11 can increase the NMMO concentration of the spin bath 11 to only a negligible

degree. In the inside bath solution 31 in the film tube 10, unless the NMMO concentration in the inside bath solution 31 is regulated, different coagulation conditions might occur, as well as a variation of the diameter of the film tube 10. Due to the constant delivery and removal of the inside bath solution 31 through the delivery and removal tube 18 and 19, respectively, a constant renewal of the inside bath solution 31 occurs, i.e., the inside bath solution 31 enriched with morpholine near the deflector roll 13 is diluted, so that the NMMO concentration of the inside bath solution 31 near the deflector roll 13 is less than or at most equal to the NMMO concentration of the spin bath 11. The tension applied to the film tube 10, together with thepressure of the spin bath 11, is enough to urge the film tube 10 against the deflector roll 13 along the line of contact 27 so that it is more or less flattened, as can be seen in Figures 1 and 2. Thus, uniform pressure conditions are established in the film tube over the entire length from just beneath the surface of the spin bath 11 to an area close to the deflector roll 13, so that the gauge or diameter of the film tube 10 is constant and shows no fluctuation or narrowing. The density of the inside bath solution 31 is thus dependent upon the throughput of the inside bath solution or morpholine solution, the amount of the inside bath and the depth of immersion of the inlet tube 18 or the point at which fresh morpholine solution is fed into the inside bath solution. The position or point at which the inside bath solution 31 flows into the film tube 10 substantially influences the gauge constancy, the level of the inside bath in the ascending film tube 10 after the deflector roll 13 and the location for the removal of the inside bath solution 31 from the interior of the film tube.

Fig. 3 shows on an enlarged scale the section indicated at A in Fig. 1. The tube 29 is lowered about 50 to 100 mm into the inside bath solution in the film tube. The aspirating tube 19 is in a position in which it establishes a level 30 inside of the tube 29, which is up to 20 mm higher or 45 mm lower than an inside bath level 28 of the inside bath solution of the film tube 10.

In other words, this means that the aspirating tube 19 assumes a position at which the inside bath solution is drawn up to a distance of as much as 20 mm higher or 45 mm lower than the tube level 28. The highest and the lowest tube levels 30 are indicated by broken lines 30_{max} and 30_{min} in Fig. 3. The aspiration usually starts below the level of the spin bath 11, so that the air section 9 situated above it and the pressure conditions there prevailing have no influence on the inside bath solution and thus cannot produce any gauge fluctuations in the film tube 10, either. If the aspiration is performed above the level of the spin bath 11, the effect of the pressure conditions in the air section 9 on the film tube 10 is negligible, since the latter is made stable in shape by the insert to the extent that it is hardly subject to any gauge variations.

Due to the adjustment of the depth of immersion of the inlet tube 18 and the constant renewal of the inside bath solution the density of the inside bath solution 31 is kept at a uniform level, which results in a constriction of the film tube 10 along the line of contact 27 with the deflector roll 13 and keeps the level of the inside bath solution 31 in the ascending film tube 10 constant for as long as desired with respect to the surface of the spin bath 11, so that irregular running and film tube gauge variations no longer occurs. The constant renewal and the minimum delivery of inside bath solution 31 are to be determined individually for each rate of extrusion or output of the film tube.

The film tube 10 emerging from the spin bath 11 then passes through precipitation and washing tubs not shown, and can also be treated with plasticizers, for example, and then dried before being wound up and further treated.

In a variation of the method, the level of the spin bath 11 inside and outside of the film tube is lowered to the upper edge of the deflector roll 13

and the film tube 10 can be sprayed inside and out with the spin bath through ring nozzles, as described, for example, in EP-A 0 006 601.

In Figures 4a and 4b, a side view and top view of the cementing system 25 are shown schematically. The cementing system 25 comprises a hollow, fixed finger 36 with a slit 37 from which the cement flowing inside of the finger 36 is applied to the longitudinal seam 38 of the tubular envelope 6. The envelope 6 is simultaneously moving vertically in the direction of the arrow to the finger 36.

The film tube 10 can be expanded alternatively or additionally to the supporting air through a tube, a ring or a spreader in flat form, free of wrinkles.

Besides the treatment of the film tube 10 in the spin bath 11, as described in Figures 1 to 4b, with complete immersion of the film tube in the spin bath, wherein the tube interior is filled with an inside bath for pressure equalization, and the inside bath level 28 can be regulated differently from the tube level 30 in the tube 29 or by the outside level of the spin bath 11, it is also possible, in the case of the previously described bilateral coating with cellulose-NMMO solution, a film of NMMO solution can be applied to the outside as well as to the inside of the film tube 10 by means of an outer and inner annular ring nozzle.

Claims

1. Method for the production of a film tube on a cellulose basis, which is strengthened by an insert, by extruding an aqueous cellulose-N-methyl-morpholine N-oxide (NMMO) solution onto the insert, characterized in that the insert is drawn from a roll, treated with emulsifiers, wetting and/or anchoring agents and formed into a tubular envelope with overlapping longitudinal seam which is cemented ahead of a nozzle block through which the envelope is brought and in which the cellulose-NMMO solution is applied to the envelope and penetrates the latter, in order to obtain an insert-reinforced film tube, that the interior of the film tube is filled with an aqueous NMMO solution, and that the film tube exits from the nozzle block and enters into a spin bath, is turned about in the latter and brought out.

2. Method according to claim 1, characterized in that the tubular envelope passes through a heating section situated ahead of the nozzle block, in which it is preheated with hot air to the temperature of the extruded cellulose-NMMO solution.

3. Method according to claim 1, characterized in that pressure-regulated supporting air is blown into the interior of the film tube after departure from the nozzle block.

4. Method according to claim 1, characterized in that the film tube is carried through a heated annular gauging disk through which a heating medium flows in a controlled circuit.

5. Method according to claim 1, characterized in that the aqueous NMMO solution is delivered through the nozzle block into the interior of the film tube

and also removed from it, the delivery and removal being performed at a distance apart from one another.

6. Method according to claim 5, characterized in that the level of the delivery of the aqueous NMMO solution is adjustable and that the removal is performed such that the level in the film tube is variably higher by up to 20 mm and lower by up to 45 mm than the level in the spin bath.

7. Method according to claim 1, characterized in that the film tube, after leaving the nozzle block, runs through an air section until it enters into the spin bath, and that in the air section an external temperature treatment takes place which regulates the rate of solidification of the cellulose-NMMO solution of the film tube.

8. Method according to claim 1, characterized in that the film tube plunges vertically into the spin bath and with maintenance of a constant tension is turned about by a powered return roll running close to the bottom of the spin bath tube and is carried out upwardly at an angle from the spin bath.

9. Method according to claim 1, characterized in that the spin bath level inside and outside of the film tube is lowered as far as the upper edge of a return roll and that the film tube is sprayed inside and out with spin bath.

10. Method according to claim 1, characterized in that the longitudinal seam of the tubular envelope is cemented with straight NMMO or a cellulose-NMMO solution at a temperature of 15 to 110°C, especially at the temperature of the cellulose-NMMO solution extruded in the nozzle block.

11. Method according to claim 1, characterized in that the cellulose content of the extruded cellulose-NMMO solution amounts to 1 to 15 wt.%, especially 3 to 7 wt.-% with respect to the total solution, and that the average degree of polymerization ranges from 250 to 800, especially from 300 to 500.

12. Method according to claim 1, characterized in that the aqueous NMMO solution of the spin bath has an NMMO concentration of 5 - 50 wt.-%, especially of 8 to 20 wt.-% and that the spin bath is adjusted to 0 to 50°C, especially 2 to 20°C.

13. Apparatus for the production of a film tube on a cellulose basis which an insert strengthens, by extrusion of an aqueous cellulose-N-methyl-morpholine N-oxide (NMMO) solution onto the insert, with a nozzle block (7) and a spin bath (11), characterized in that a supply roll (2) for the insert (3), a deflector roll (4) with a system for the application of additives onto the insert carried from the supply roll over the deflector roll, and a forming section (5) in which the insert (3) is formed into a tubular envelope (6) with overlapping longitudinal seam are present, that the tubular envelope (6) passes through the nozzle block (7) which is preceded by an adhering system (25) for cementing the longitudinal seam of the tubular envelope (6) and which contains an annular nozzle (21) from whose nozzle gap the cellulose-NMMO solution is extruded into the tubular envelope (6) to form a film tube (10), that between the exit from the nozzle block (7) and the spin bath (11) a controlled-temperature air section (9) is present in a spin tub (12), that near the bottom of the spin tub (12) a deflector roll (13) is disposed for the film tube plunging vertically into the spin bath, and that a delivery and removal tube (18, 19) is present in the interior of the film tube (19) for the aqueous NMMO solution, as well as a duct (20) for the supporting air.

14. Apparatus for the production of a film tube according to claim 13, characterized in that the insert (3) is selected from the group, paper, nonwoven, fiber fleece, fiber paper, the fibers being especially long hemp fibers.

15. Apparatus for the production of a film tube according to claim 13, characterized in that a preheating system (15) for the tubular envelope (6) is disposed ahead of the nozzle block (7), and that the preheating system (15) is connected via hot air duct (22, 23) and an exhaust duct (24) to a controllable heater (17) from which air heated in the circuit flows into the preheating system (15) and from which cooled air flows back into the heater (17).

16. Apparatus for the production of a film tube according to claim 13, characterized in that the nozzle block (7) contains a ring nozzle (21) which is heated by a heating medium, and that the delivery tube, the removal tube (18, 19) and the duct (20) for the air supporting the film tube (10) are brought centrally through a gauging ring disk (8) which is arranged concentrically with the ring nozzle (21) in the film tube interior and forms with the latter an annular gap (26) through which the film tube (10) runs.

17. Apparatus for the production of a film tube according to claim 16, characterized in that the gauging ring disk (8) is connected to the heating circuit (16) for the purpose of heating.

18. Apparatus for the production of a film tube according to claim 16, characterized in that the delivery tube (18) and the removal tube (19) are individually height-adjustable within the film tube (10).

19. Apparatus for the production of a film tube according to claim 18, characterized in that the delivery tube (18) is disposed in an upper position at the beginning of the delivery of the aqueous NMMO solution into the film tube (10) and at the

start of continuous operation assumes a position above the return roll (13).

20. Apparatus for the production of a film tube according to claim 16, characterized in that the heating medium flows through the ring nozzle and is carried in a controlled heating circuit (16).

21. Apparatus for the production of a film tube according to claim 13, characterized in that the air section (9) amounts to 1 to 1000 mm, especially 200 to 500 mm, and that if necessary the film tube (10) can be heated to delay its solidification or cooled to accelerate its solidification in the air section.

22. Apparatus for the production of a film tube according to claim 13, characterized in that the return roll (13) disposed near the bottom of the spin tub (12) is driven and exerts a constant tension on the vertically descending film tube (10).

23. Apparatus for the production of a film tube according to claim 22, characterized in that the film tube (10) lies flat against the return foll (13) along a line of contact (27) as a result of the tension exerted on the film tube (10).

24. Apparatus for the production of a film tube according to claim 13, characterized in that the spin bath (11) and the aqueous NMMO solution in the film tube (10) have equal NMMO concentrations at the beginning of the extrusion of the film tube (10).

25. Apparatus for the production of a film tube according to claim 13, characterized in that the excess pressure of the supporting air in the film tube (10) amounts to 0.1 to 10 mbar in the range of the air section (9).

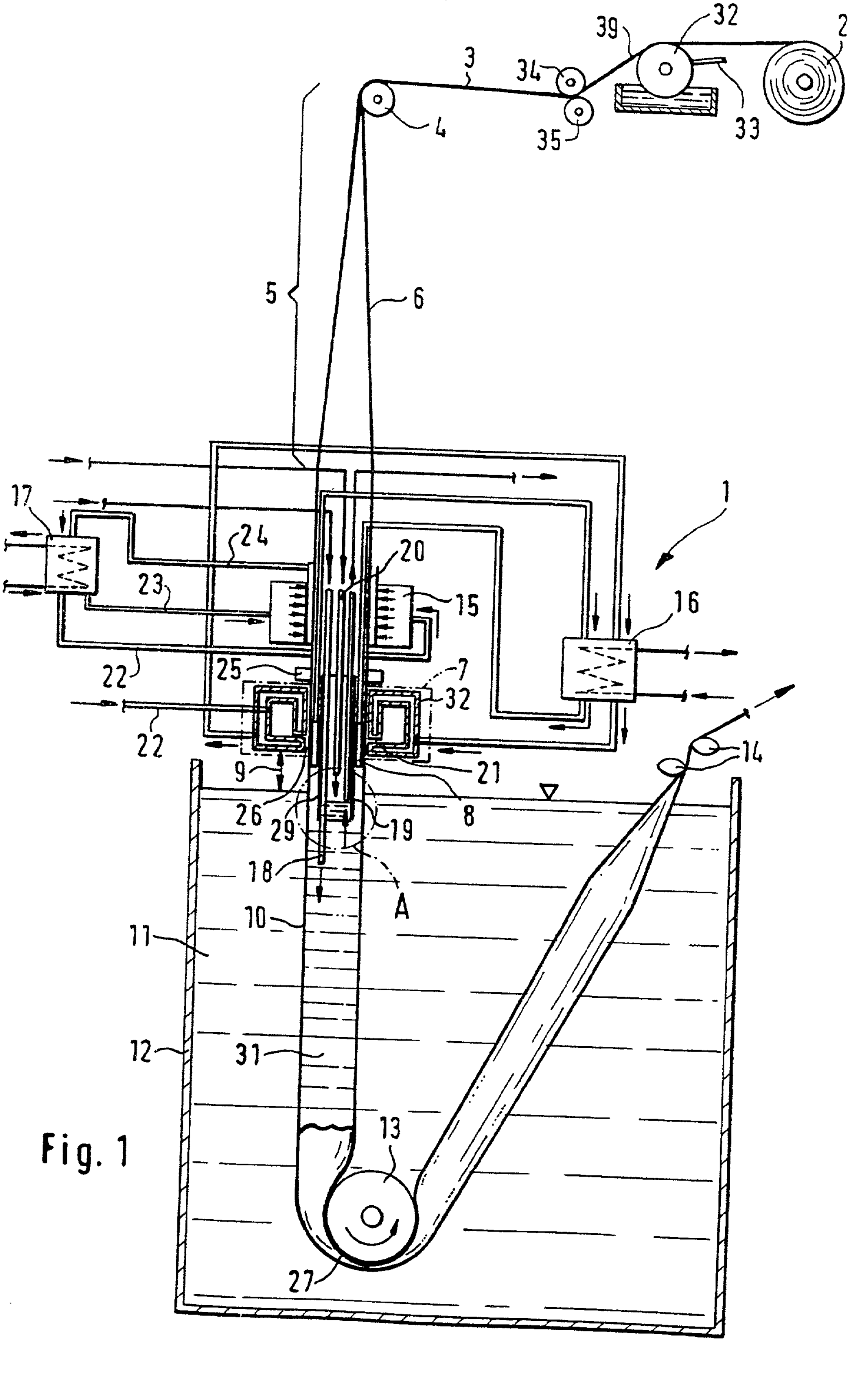
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22
23
24
25
26
27
29
31
32
33
34
35
39
A

Fig. 1

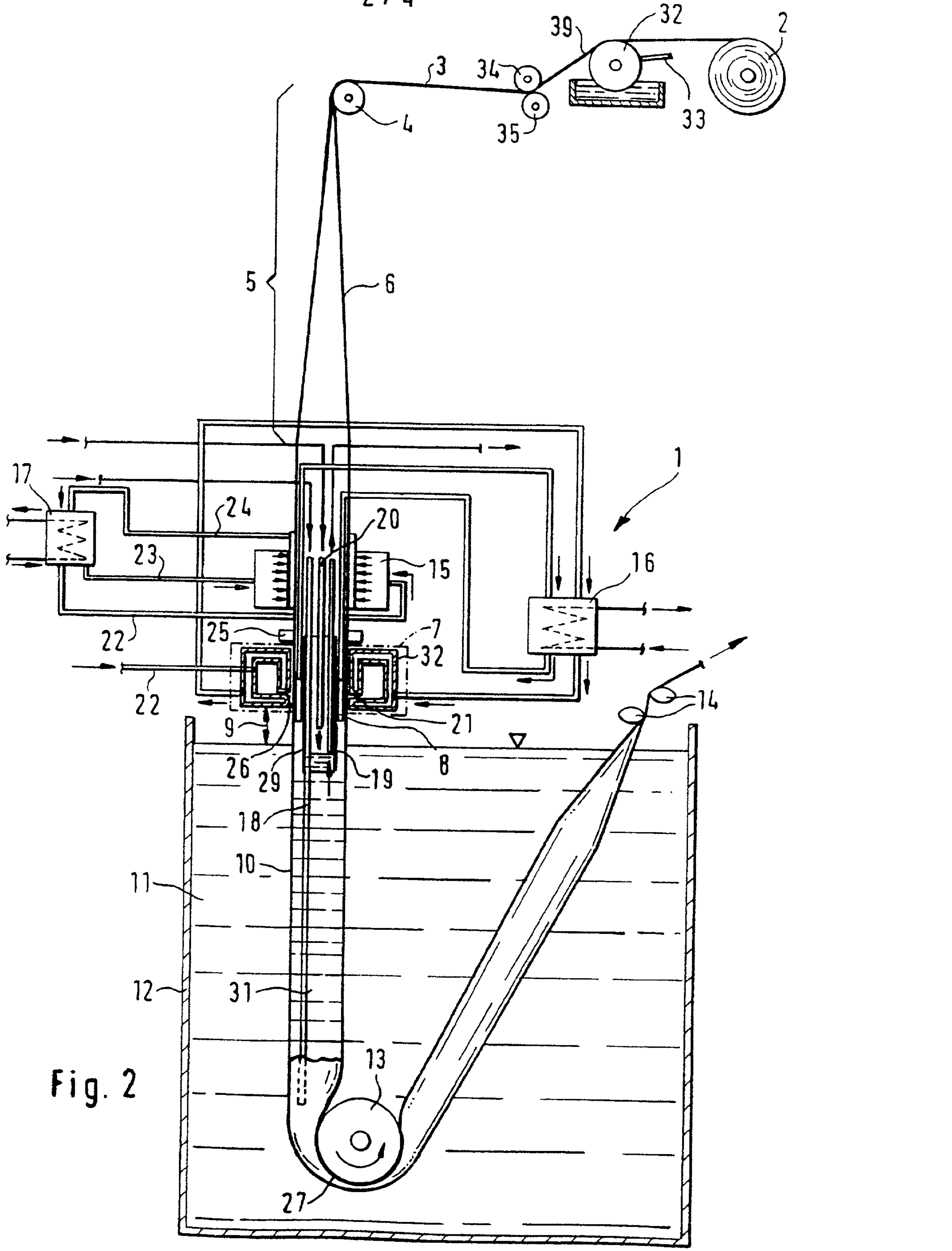
39
32
2
3
34
4
35
33
5
6
1
17
24
20
23
15
16
22
25
7
32
22
9
21
14
26
29
19
8
18
10
11
12
31
13
27

Fig. 2

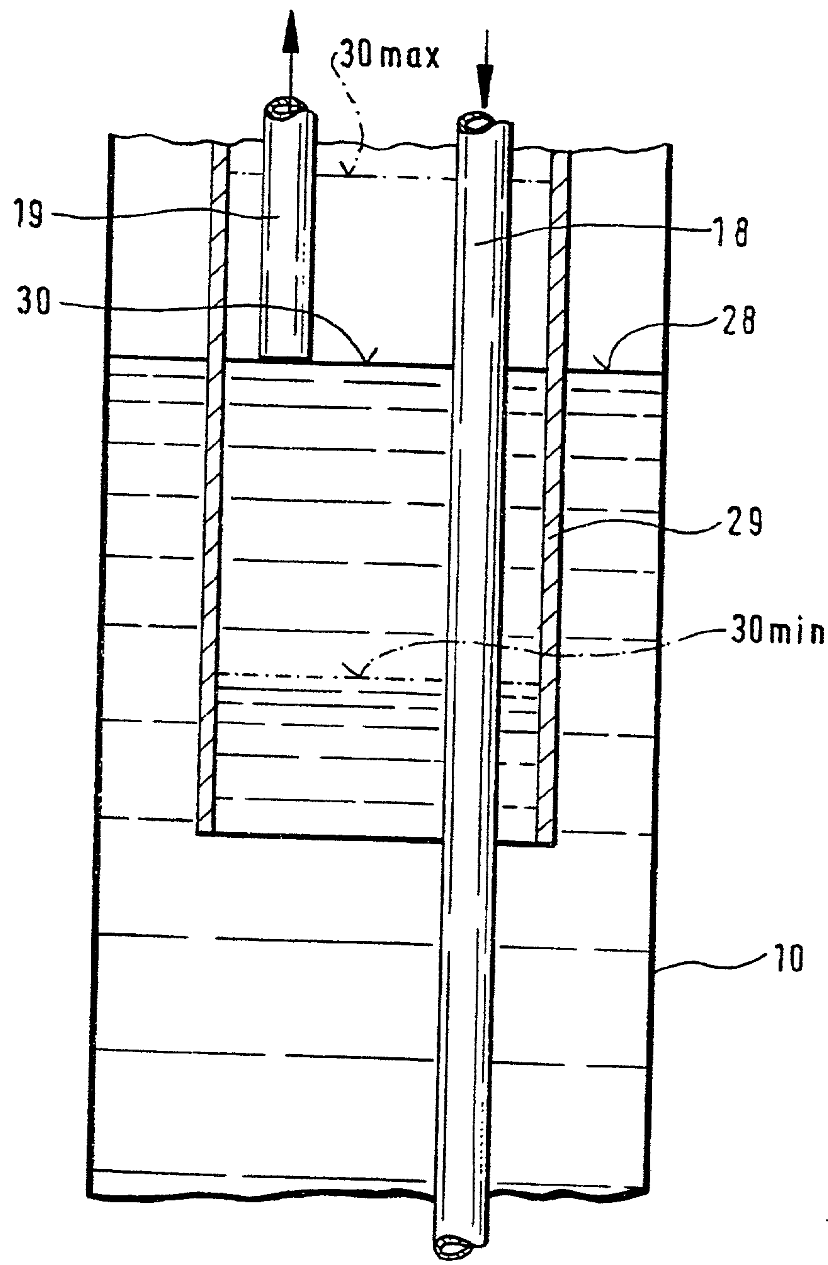

Fig. 3

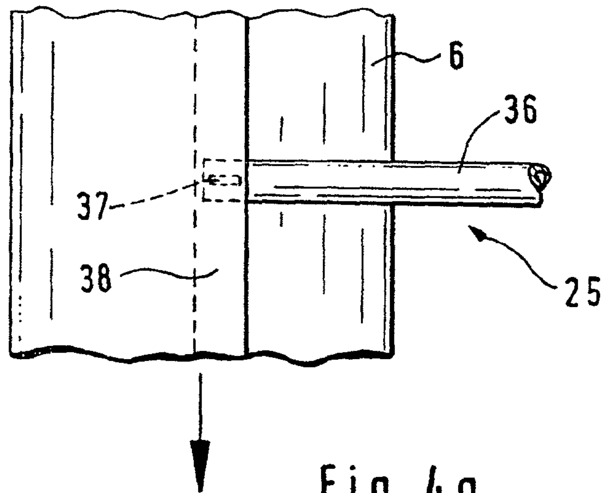

Fig. 4a

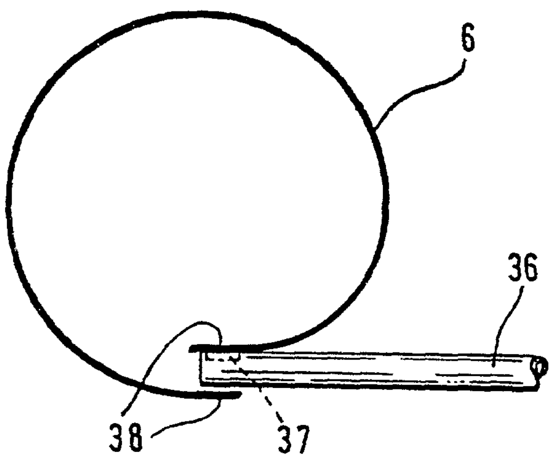

Fig. 4b

Attorney Docket No: 37838-0009

DECLARATION AND POWER OF ATTORNEY

As a below named inventor, I HEREBY DECLARE:

THAT my residence, post office address, and citizenship are as stated below next to my name:

THAT I believe I am the original, first, and sole inventor (if only one inventor is named below) or an original, first, and joint inventor (if plural inventors are named below or in an attached Declaration) of the subject matter which is claimed and for which a patent is sought on the invention entitled

Method and Apparatus for the Production of a Film Tube on a Cellulose Basis

the specification of which (check one)

____ is attached hereto.

XX was filed on March 22, 2001 as United States Application Number ~~or PCT International Application Number~~ 09/787,416 and was amended on ______________ (if applicable).

THAT I do not know and do not believe that the same invention was ever known or used by others in the United States of America, or was patented or described in any printed publication in any country, before I (we) invented it;

THAT I do not know and do not believe that the same invention was patented or described in any printed publication in any country, or in public use or on sale in the United States of America, for more than one year prior to the filing date of this United States application;

THAT I do not know and do not believe was first patented or made the subject of an inventor's certificate that issued in any country foreign to the United States of America before the filing date of this United States application if the foreign application was filed by me (us), or by my (our) legal representatives or assigns, more than twelve months (six months for design patents) prior to the filing date of this United States application;

THAT I have reviewed and understand that contents of the above-identified specification, including the claim(s), as amended by any amendment specifically referred to above;

THAT I believe that the above-identified specification contains a written description of the invention, and of the manner and process of making and using it, in such full, clear, concise, and exact terms as to enable any person skilled in the art to which it pertains, or with which it is most nearly connected, to make and use the invention, and sets forth the best mode contemplated by me of carrying out the invention; and

THAT I acknowledge the duty to disclose to the U.S. Patent and Trademark Office all information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, §1.56.

I HEREBY CLAIM foreign priority benefits under Title 35, United States Code §119(a)-(d) of §365(b) of any foreign application9s) for patent or inventor's certificate, or §365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below any foreign application for patent or inventor's certificate or of any PCT international application having a filing date before that of the application on which priority is claimed.

Attorney Docket No: 37838-0009

Prior Foreign Application Number	Country	Foreign Filing Date	Priority Claim?	Certified Copy Attached?
198 43 723.4	GERMANY	September 24, 1998	YES	NO

I HEREBY CLAIM the benefit under Title 35, United States Code §119(e) of any United States provisional application(s) listed below.

U.S. Provisional Application Number	Filing Date

I HEREBY CLAIM the benefit under Title 35, United States Code, §120 of any United States application(s), or §365(c) of any PCT International application designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of Title 35, United States Code §112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, §1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application.

U.S. Patent Application Number	PCT Patent Application Number	Patent Filing Date	Parent Patent Number
	PCT/EP99/06820		

I HEREBY APPOINT the following registered attorneys and agents of the law firm of Heller Ehrman White & McAuliffe LLP to have full power to prosecute this application and any continuations, divisions, reissues, and reexaminations thereof, to receive the patent, and to transact all business in the United States Patent and Trademark Office connected therewith:

Ronald J. Kamis	Reg. No.	41,104
Susan E. Shaw McBee	Reg. No.	39,294
Marvin Motsenbocker	Reg. No.	36,614
Colin G. Sandercock	Reg. No.	31,298

and I request that all correspondence be directed to:

Susan E. Shaw McBee
Heller Ehrman White & McAuliffe
815 Connecticut Avenue, N.W. Suite 200
Washington, D.C. 20006-4004
(202) 263-8900 / (202) 785 8877

I UNDERSTAND AND AGREE THAT the foregoing attorneys and agents appointed by me to prosecute this application do not personally represent me or my legal interests, but instead represent the interests of the legal owner(s) of the invention described in this application.

I FURTHER DECLARE THAT all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so make are punishable by a find or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

100

Name of First inventor	Herbert GORD
jResidence	Ahornweg 5, D-55218 Ingelheim, Federal Republic of Germany
Citizenship	German DEX
Post Office Address	Ahornweg 5, D-55218 Ingelheim, Federal Republic of Germany
Inventor's signature	Herbert Gord
Date	March 29, 2001

200

Name of the Second Inventor	Dr. Klaus-Dieter HAMMER, Ph.D.
Residence	An der Hasenquelle 25, D-55120 Mainz, D-Federal Republic of Germany
Citizenship	German DEX
Post Office Address	An der Hasenquelle 25, D-55120 Mainz, D-Federal Republic of Germany
Inventor's signature	Klaus-Dieter Hammer
Date	March 29, 2001

300

Name of the Third Inventor	Helmut SATTLER
Residence	Freudenbergstrasse 36, D-65201 Wiesbaden, Federal Republic of Germany
Citizenship	German DEX
Post Office Address	Freudenbergstrasse 36, D-65201 Wiesbaden, Federal Republic of Germany
Inventor's signature	Helmut Sattler
Date	March 29, 2001

400

Name of the Fourth Inventor	Dr. Rainer NEEFF, Ph.D.
Residence	Am Schlosspark 85, D-65203 Wiesbaden, Federal Republic of Germany
Citizenship	German DEX
Post Office Address	Am Schlosspark 85, D-65203 Wiesbaden, Federal Republic of Germany
Inventor's signature	Rainer Neeff
Date	March 29, 2001